In situ environmental transmission electron microscopy investigation of core-shell supported co-catalyst system for optimized visible-light water splitting

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As a result of diminishing fossil fuel reserves, there is an increasing need to switch energy dependence to renewable resources such as sunlight. Photocatalysts provide a viable route for converting solar energy into chemical bonds. In order to optimize the performance of such materials, a fundamental understanding of their reaction mechanisms, chemical behavior, structure and morphology before, during and after reaction using in situ investigations is necessary. Here, we focus on the in situ characterization of photocatalysts [1] in an environmental transmission electron microscope (ETEM) [2,3]. Such fundamental insight can be used for further material optimization with respect to performance and stability [4].

The activity of photocatalysts for water splitting under visible light exposure can be enhanced by adding co-catalysts for the hydrogen evolution reaction (HER) on their surface, Pt being one of the most commonly used. Pt is a reversible catalyst for HER, meaning that unless precautions are taken it will also efficiently catalyze the reverse reaction, resulting in a loss of net activity. The addition of a shell on top of the co-catalyst has proven an efficient method to dramatically reduce the reverse reaction in both catalytic [5] and photocatalytic systems [6]. Encapsulated Pt is obtained by substrate reduction in H2 and is a novel promising system for enhanced water splitting using visible light.

In this work the encapsulation of supported co-catalysts on a number of semiconductor substrates including SrTiO3 and TiO2 is studied using ETEM techniques (Fig.1). The shell formation mechanism is studied in situ as a function of reaction conditions such as temperature, pressure, time, and atmosphere composition. Catalytic efficiency of the encapsulated samples is tested in parallel using micro-reactors [7] allowing for direct correlation of reaction conditions and catalyst performance.

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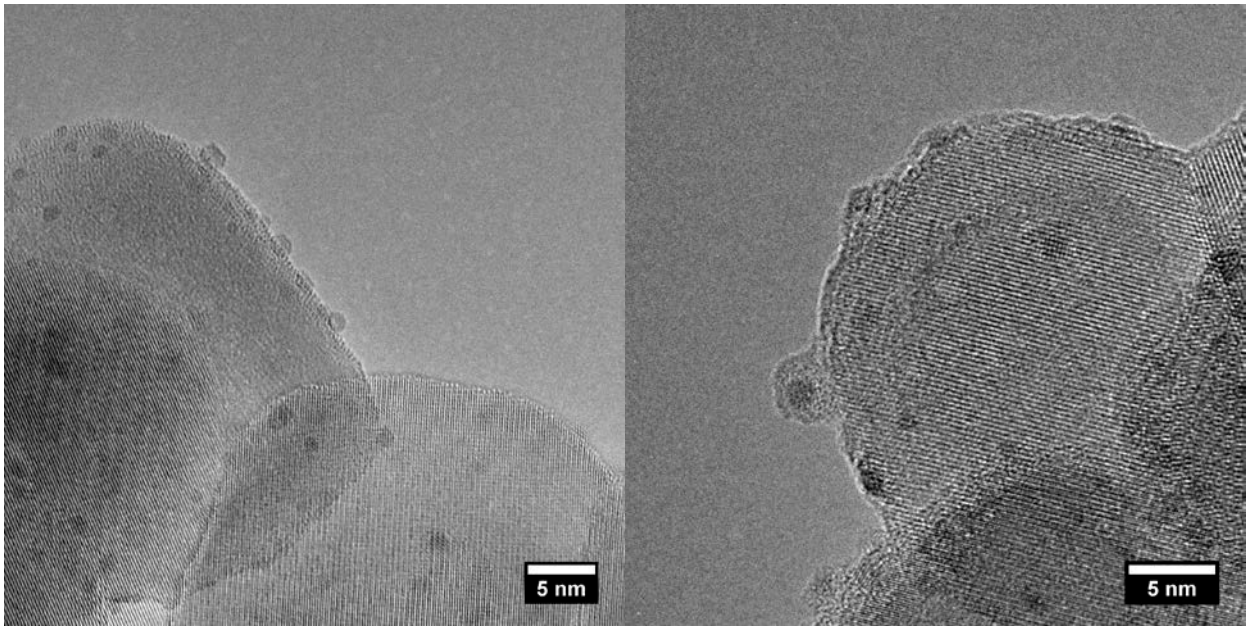

Figure 1. *Bright field TEM images of STO-supported Pt nanoparticles before (left) and after (right) high-temperature in situ reduction in H_2.*